

PATENT ABSTRACTS OF JAPAN

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(54) METHOD FOR MODIFYING POLYPROPYLENE

(57)Abstract:

PROBLEM TO BE SOLVED: To enable PP to be efficiently modified by bringing it into contact with a polar monomer in the presence of a peroxide in a recovered solvent having a pH of 6.5 or higher.

SOLUTION: PP is modified by bringing it into contact with a polar monomer (pref. an unsatd. carboxylic anhydride) in the presence of a peroxide in a solvent (pref. a halohydrocarbon compd.). The solvent used here is a recovered one having a pH of 6.5 or higher and may be one obtd. by purifying a recovered solvent by distillation and bringing the purified one into contact with a basic solid. Thus PP can be efficiently modified.

LEGAL STATUS

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CLAIMS

[Claim(s)]

[Claim 1] The qualification approach of the polypropylene characterized by using that whose PH is moreover 6.5 or more with the solvent collected as a solvent in the approach of contacting the bottom of existence of a peroxide in a solvent in polypropylene and a polar-group content monomer, and embellishing polypropylene.

[Claim 2] The approach according to claim 1 a polar-group content monomer is an unsaturated-carboxylic-acid anhydride, and a solvent is a halogenated hydrocarbon compound.

[Claim 3] The approach according to claim 1 using what carried out distillation purification of the solvent collected as a solvent, carried out contact processing with the further basic solid-state, and was obtained.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the qualification approach of polypropylene. It is related with the approach using the recovery solvent with which it is satisfied of specific conditions as a solvent used in detail.

[0002]

[Description of the Prior Art] Polypropylene is lightweight, is the cheap polymer which was moreover excellent in rigidity, and is used for various applications. However, since permeability and steam permeability are large, using by making it the resin and the multilayer containing others, resin, especially a polar group is performed. Since it faces making it a multilayer and the adhesive property of polypropylene and other resin is inferior, it is performed widely that what carried out the graft of the unsaturated carboxylic acid, such as a maleic anhydride and itaconic acid anhydride, and embellished it in polypropylene uses as resin for glue lines.

[0003] Although it is possible in the various condition, such as a melting condition, a solution condition, and a slurry regime, as conditions at the time of carrying out the graft of the unsaturated carboxylic acid, what was carried out in the state of the solution as engine performance of the embellished polypropylene which is obtained is most excellent.

[0004]

[Problem(s) to be Solved by the Invention] however, the approach of reacting in the state of a solution needs recovery utilization of a solvent -- etc. -- when the collected solvent is used for an economically disadvantageous top, the problem that graft efficiency is bad etc. is in it.

[0005]

[Means for Solving the Problem] this invention persons considered wholeheartedly the qualification approach of the polypropylene which solved the above-mentioned technical problem, and completed this invention.

[0006] That is, this invention is the qualification approach of the polypropylene moreover characterized by using that whose PH is 6.5 or more with the solvent collected as a solvent in the approach of contacting the bottom of existence of a peroxide in a solvent in polypropylene and a polar-group content monomer, and embellishing polypropylene.

[0007]

[Embodiment of the Invention] As polypropylene used in this invention, not only the homopolymer of a propylene but random or a block copolymer can manufacture by usable various approaches, and is available in a commercial scene again. Crystalline polypropylene is desirable in respect of machine physical properties, an adhesive property, etc. It can use, even if it is not only isotactic polypropylene but syndiotactic polypropylene as here crystalline polypropylene. That whose rate of the raceme pentad measured by ^{13}C -NMR as a scale of crystalline polypropylene or a meso pentad is 0.7 or more can use preferably.

[0008] What combined the metallocene compound which has the catalyst system which uses as a transition metal catalyst component the titanium tetrachloride supported with the titanium trichloride by which conversion was carried out with the electron-donative compound, or the electron-donative compound by the magnesium chloride by which conversion was carried out if

considered as the manufacture catalyst of these polypropylene, and consists of an electron-donative compound trialkylaluminium or dialkyl aluminum chloride, and if needed, or the restrained ligand, aluminoxane or the metallocene compound, the organometallic compound, and the compound that can generate a stable anion can be illustrated.

[0009] It is applicable also as a polymerization method by any polymerization methods, such as a solvent polymerization method, a bulk-polymerization method, and a vapor-phase-polymerization method.

[0010] It expresses with the limiting viscosity number measured with 135-degree-C tetralin solution from points, such as viscosity of the solution in the case of qualification, as molecular weight of the polypropylene used in this invention, and they are about 0.05-1.5 dl/g preferably 0.01 to 3.0 dl/g.

[0011] As a polar-group content monomer used in this invention, unsaturated carboxylic acid can especially use preferably, and an unsaturated-carboxylic-acid anhydride is used. Specifically An acrylic acid, a methacrylic acid, a maleic acid, boletic acid, Unsaturated carboxylic acid, such as an itaconic acid, a citraconic acid, a tetrahydrophthalic acid, the bicyclo (2, 2, 1) hept-2-en -5, and 6-dicarboxylic acid, A maleic anhydride, itaconic acid anhydride, an anhydrous citraconic acid, tetrahydro phthalic anhydride, The anhydride of unsaturated carboxylic acid, such as the bicyclo (2, 2, 1) hept-2-en -5 and 6-dicarboxylic acid anhydride, Unsaturated-carboxylic-acid ester, such as a methyl acrylate, a methyl methacrylate, ethyl methacrylate, methacrylic-acid butyl, maleic-acid dimethyl, maleic-acid monomethyl, boletic acid dimethyl, itaconic-acid dimethyl, and citraconic-acid dimethyl, etc. is used.

[0012] if it is the thing of a comparatively inactive high-boiling point to a radical as a solvent used in this invention -- available -- a saturated hydrocarbon compound and an aromatic hydrocarbon compound -- desirable -- a halogenated hydrocarbon compound -- it is an aromatic series halogenated hydrocarbon compound especially preferably. Specifically A hexane, a heptane, an octane, Decane, a dodecane, tetradecane, Aliphatic hydrocarbon compounds, such as kerosene, methylcyclopentane, a cyclohexane, Alicyclic group hydrocarbon compounds, such as a methylcyclohexane, cyclooctane, and a cyclo dodecane, Benzene, toluene, a xylene, ethylbenzene, a cumene, ethyl toluene, Aromatic hydrocarbon compounds, such as trimethyl benzene, a cymene, and diisopropylbenzene, Halogenated hydrocarbon compounds, such as a chlorobenzene, a bromobenzene, O-dichlorobenzene, a carbon tetrachloride, trichloroethane, a trichloroethylene, tetrachloroethane, and tetrachloroethylene, etc. are illustrated.

[0013] In this invention, recovery reuse of the above-mentioned solvent is carried out. It is controlling so that PH of the solvent with which it faced carrying out recovery reuse of the solvent, and important one's was collected becomes 6.5 or more. PH of a solvent is contacted in 100ml of water to 200ml of solvents, and it is expressed with PH of a water layer PH here. Usually, the embellished polypropylene is separated by making it deposit from a solvent in a reaction solution, and filtering by adding the poor solvent of polypropylene, in it. It dissociates from the poor solvent of polypropylene etc. by usually distilling a solvent from the solution of a filtrate. Although it is possible, after distilling preferably and removing the poor solvent of polypropylene, as for controlling PH also by performing distillation actuation strictly or processing a recovery solvent with an alkali solution, it is efficient to carry out by contacting a basic solid-state. Especially although there is nothing especially as an upper limit of PH here, carrying out to 9.0 or less is [10.0 or less] desirable.

[0014] the inorganic solid base as a here basic solid-state, and an organic solid base -- although either is usable -- from the adsorption capacity force of an acid -- thinking -- an inorganic solid base -- concrete -- the hydroxide of alkali metal and alkaline earth metal, and carbide -- the complex salt of them etc. can use preferably. Specifically MgO, aluminum(OH)3xH2O, and Mg(OH)2, Mg(OH)2aluminum(OH)3xCO3yH2O and aluminum(OH)3, A compound basic oxide, a hydroxide, etc. which use aluminum(OH)3NaHCO3, Mg6aluminum2(OH)16CO34H2O, Mg4.5aluminum2(OH)13CO33.5H2O, Mg, aluminum, Si, etc. as a principal component can be illustrated.

[0015] In this invention, peroxide or a hydroperoxide is preferably used as a peroxide, and, specifically, there are alkyl peroxide, aryl peroxide, acyl peroxide, aroyl peroxide, ketone peroxide,

peroxy carbonate, peroxy carboxylate, a hydroperoxide, etc. As alkyl peroxide, they are diisopropyl peroxide and G. tert-butyl peroxide, 2, 5-dimethyl - 2 Five - As aryl peroxide, such as G tert-butylperoxy hexyne, a methyl-ethyl-ketone hydroperoxide, cyclohexanone peroxide, etc. are raised as ketone peroxide, such as dibenzoyl peroxide, as aroyl peroxide, such as dilauryl peroxide, as acyl peroxide, such as dicumyl peroxide. As a hydroperoxide, it is tert. - A butyl hydroperoxide, cumene hydroperoxide, etc. can be mentioned. In these, it is G tert-butyl peroxide, 2, and 5-dimethyl. - 2 Five - G tert-butylperoxy hexyne -3, dicumyl peroxide, dibenzoyl peroxide, etc. are desirable.

[0016] In this invention, it is the temperature which polypropylene dissolves in a solvent and a peroxide decomposes at a suitable rate as a reaction condition at the time of embellishing polypropylene, and it is common to carry out time until a peroxide will decompose substantially. As long as it is held as reaction pressure at the above-mentioned temperature, as for a limit, it is desirable to carry out under an inert gas ambient atmosphere so that the radical generated although there was nothing may not be consumed vainly.

[0017]

[Example] An example is shown below and this invention is further explained to it.

[0018] Polypropylene 159.3g of 300g / 10min and 22.9g of maleic anhydrides were added to mono-chlorobenzene 792g, and at 125 degrees C, MFI (they are measurement and ASTM-D1238 in 2.16kg of loads at 230 degrees C) which processed polypropylene Mitsui no BUREN JS-G by example of reference 1 Mitsui Toatsu Chemicals, Inc. with 2 by Nippon Oil & Fats Co., Ltd., the 5-dimethyl -2, and 5-G tert-butylperoxy hexyne -3, and obtained it with the extruder heated for 1 hour, and dissolved. Next, in addition, it reacted at 125 more degrees C for 2 hours, having covered the solution which dissolved dicumyl peroxide 22.8g in mono-chlorobenzene 169g over the above-mentioned solution for 5 hours. In addition, it was 7.0 when PH of the mono-chlorobenzene used here was measured. Measurement of PH was performed here by contacting 100ml water in mono-chlorobenzene 200ml, and measuring PH of water.

[0019] The reaction solution was lowered to 25 degrees C, and the polypropylene which added acetone 1000ml and was embellished was deposited. Subsequently, it filtered and the embellished polypropylene 1525g of filtrates and whose dry weight are 165g was obtained. The limiting viscosity number measured with the 135-degree C tetralin solution of the obtained polymer was 0.28 dl/g, and the maleic-anhydride content was 10.9 % of the weight.

[0020] The filtrate collected in the example 1 of example 1 reference was distilled in batch with the reflux ratio 10 using the distillation apparatus of ten steps of theoretical plates. Under the present circumstances, it was as **** and could be 20% as a still residue 60% 20% as a fore run among the fractions after temperature becomes 109 degrees C. It was 7.0 when PH of this fraction was measured like the example 1 of reference. When the same experiment as the example 1 of reference was conducted using this fraction as a solvent, the limiting viscosity number was 0.28 dl/g and the maleic-anhydride content was 11.2 % of the weight.

[0021] When the mono-chlorobenzene which set example of comparison 1 reflux ratio to 0.1, and was collected as 10% (PH of this fraction was 5.5.) as a still residue 80% as **** 10% as a fore run was used and also having been carried out like the example 1, the limiting viscosity number was 0.35 dl/g and the maleic-anhydride content was 8.0 % of the weight.

[0022] PH was 6.8 when contact processing (they are the two sections to the solvent 100 section) of this fraction used in the example 1 of example 2 comparison was carried out by KYOU WORD 115 (MgO;97.11% and specific-surface-area;100m2 / g) made from Consonance Chemical industry. When experimented like the example 1 of reference using this solvent, the limiting viscosity number was 0.28 dl/g and the maleic-anhydride content was 11.0 % of the weight.

[0023]

[Effect of the Invention] It is extremely worthy that it is possible to embellish polypropylene efficiently and industrially by enforcing the approach of this invention.

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TECHNICAL FIELD

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PRIOR ART

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[0003] Although it is possible in the various condition, such as a melting condition, a solution condition, and a slurry regime, as conditions at the time of carrying out the graft of the unsaturated carboxylic acid, what was carried out in the state of the solution as engine performance of the embellished polypropylene which is obtained is most excellent.

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TECHNICAL PROBLEM

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MEANS

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[Embodiment of the Invention] As polypropylene used in this invention, not only the homopolymer of a propylene but random or a block copolymer can manufacture by usable various approaches, and is available in a commercial scene again. Crystalline polypropylene is desirable in respect of machine physical properties, an adhesive property, etc. It can use, even if it is not only isotactic polypropylene but syndiotactic polypropylene as here crystalline polypropylene. That whose rate of the raceme pentad measured by ^{13}C -NMR as a scale of crystalline polypropylene or a meso pentad is 0.7 or more can use preferably.

[0008] What combined the metallocene compound which has the catalyst system which uses as a transition metal catalyst component the titanium tetrachloride supported with the titanium trichloride by which conversion was carried out with the electron-donative compound, or the electron-donative compound by the magnesium chloride by which conversion was carried out if considered as the manufacture catalyst of these polypropylene, and consists of an electron-donative compound trialkylaluminium or dialkyl aluminum chloride, and if needed, or the restrained ligand, aluminoxane or the metallocene compound, the organometallic compound, and the compound that can generate a stable anion can be illustrated.

[0009] It is applicable also as a polymerization method by any polymerization methods, such as a solvent polymerization method, a bulk-polymerization method, and a vapor-phase-polymerization method.

[0010] It expresses with the limiting viscosity number measured with 135-degree-C tetralin solution from points, such as viscosity of the solution in the case of qualification, as molecular weight of the polypropylene used in this invention, and they are about 0.05-1.5 dl/g preferably 0.01 to 3.0 dl/g.

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[0012] if it is the thing of a comparatively inactive high-boiling point to a radical as a solvent

used in this invention -- available -- a saturated hydrocarbon compound and an aromatic hydrocarbon compound -- desirable -- a halogenated hydrocarbon compound -- it is an aromatic series halogenated hydrocarbon compound especially preferably. Specifically A hexane, a heptane, an octane, Decan, a dodecane, tetradecane, Aliphatic hydrocarbon compounds, such as kerosene, methylcyclopentane, a cyclohexane, Alicyclic group hydrocarbon compounds, such as a methylcyclohexane, cyclooctane, and a cyclo dodecane, Benzene, toluene, a xylene, ethylbenzene, a cumene, ethyl toluene, Aromatic hydrocarbon compounds, such as trimethyl benzene, a cymene, and diisopropylbenzene, Halogenated hydrocarbon compounds, such as a chlorobenzene, a bromobenzene, O-dichlorobenzene, a carbon tetrachloride, trichloroethane, a trichloroethylene, tetrachloroethane, and tetrachloroethylene, etc. are illustrated.

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[0014] the inorganic solid base as a here basic solid-state, and an organic solid base -- although either is usable -- from the adsorption capacity force of an acid -- thinking -- an inorganic solid base -- concrete -- the hydroxide of alkali metal and alkaline earth metal, and carbide -- the complex salt of them etc. can use preferably. Specifically MgO, aluminum(OH) $3 \times H_2O$, and Mg(OH) $_2$, Mg(OH) $_2$ aluminum(OH) $3 \times CO_3 \times H_2O$ and aluminum(OH) $_3$, A compound basic oxide, a hydroxide, etc. which use aluminum(OH) $_3NaHCO_3$, Mg $_6$ aluminum $_2$ (OH) $16CO_3 \times 4H_2O$, Mg $_4.5$ aluminum $_2$ (OH) $13CO_3 \times 3.5H_2O$, Mg, aluminum, Si, etc. as a principal component can be illustrated.

[0015] In this invention, peroxide or a hydroperoxide is preferably used as a peroxide, and, specifically, there are alkyl peroxide, aryl peroxide, acyl peroxide, aroyl peroxide, ketone peroxide, peroxy carbonate, peroxy carboxylate, a hydroperoxide, etc. As alkyl peroxide, they are diisopropyl peroxide and G. tert-butyl peroxide, 2, 5-dimethyl - 2 Five - As aryl peroxide, such as G tert-butylperoxy hexyne, a methyl-ethyl-ketone hydroperoxide, cyclohexanone peroxide, etc. are raised as ketone peroxide, such as dibenzoyl peroxide, as aroyl peroxide, such as dilauryl peroxide, as acyl peroxide, such as dicumyl peroxide. As a hydroperoxide, it is tert. - A butyl hydroperoxide, cumene hydroperoxide, etc. can be mentioned. In these, it is G tert-butyl peroxide, 2, and 5-dimethyl. - 2 Five - G tert-butylperoxy hexyne -3, dicumyl peroxide, dibenzoyl peroxide, etc. are desirable.

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EXAMPLE

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[0019] The reaction solution was lowered to 25 degrees C, and the polypropylene which added acetone 1000ml and was embellished was deposited. Subsequently, it filtered and the embellished polypropylene 1525g of filtrates and whose dry weight are 165g was obtained. The limiting viscosity number measured with the 135-degree C tetralin solution of the obtained polymer was 0.28 dl/g, and the maleic-anhydride content was 10.9 % of the weight.

[0020] The filtrate collected in the example 1 of example 1 reference was distilled in batch with the reflux ratio 10 using the distillation apparatus of ten steps of theoretical plates. Under the present circumstances, it was as **** and could be 20% as a still residue 60% 20% as a fore run among the fractions after temperature becomes 109 degrees C. It was 7.0 when PH of this fraction was measured like the example 1 of reference. When the same experiment as the example 1 of reference was conducted using this fraction as a solvent, the limiting viscosity number was 0.28 dl/g and the maleic-anhydride content was 11.2 % of the weight.

[0021] When the mono-chlorobenzene which set example of comparison 1 reflux ratio to 0.1, and was collected as 10% (PH of this fraction was 5.5.) as a still residue 80% as **** 10% as a fore run was used and also having been carried out like the example 1, the limiting viscosity number was 0.35 dl/g and the maleic-anhydride content was 8.0 % of the weight.

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